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(54) Title: APPARATUS FOR ATOMIZING A LIQUID PRODUCT

(57) Abstract: This invention relates to an apparatus for atomizing a liquid product using a propellant, which may be integrated into aerosol packs, for atomization of a liquid product. The liquid product may have a high viscosity. The total flow rate ranges from 0.5 grams per second to 0.01 grams per second through a single capillary tube. The liquid product is atomised within a capillary tube. The apparatus may be designed as a handheld unit or as a stationary or mobile unit using a plurality of capillary tubes.



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Apparatus for atomizing a liquid product

This invention relates to an apparatus for atomizing a liquid product which can be integrated into aerosol packs, which may be pre-pressurized. Such an apparatus may be integrated into a spray can, which is operable by simply pushing a closure mechanism to open valves for dispensing the contents of the can.

A generic apparatus for atomizing a liquid product uses pressure from a propellant, contained within a storage container connected thereto or alternatively a pump to pressurize the storage container. Such known devices use a tube to transport the liquid product to be atomized to an atomizing nozzle where droplets are formed from the liquid product. In order to effectively atomize a liquid product by a conventional atomizing apparatus, comparatively large volumes of propellant, dilutant and/or solvent, in relation to the liquid product are necessary, both for providing sufficient pressure for the atomization process and for reducing the viscosity of the liquid product, which forms the actual active ingredient of the system. The propellant is conventionally used in a volumetric ratio of 2000 : 1 to 20.000 : 1 of gas to liquid product, when determined at atmospheric pressure. The propellant may be compressed air, nitrogen, or, conventionally a volatile organic compound such as butane and chlorinated or fluorinated hydrocarbons, which are liquid in a compressed state.

For the purposes of this disclosure, the term "liquid product" refers to a composition which is liquid at room temperature, containing the active ingredient, which is formulated as a solution, suspension, or dispersion, like e.g. hairspray, a paint composition etc., containing the dilutant only necessary for formulating the active ingredient like soluble resins or dispersible particles for e.g. paints or hairspray, without necessarily incorporating additional dilutants in admixture. In conventional systems, this liquid product has to be diluted further by additional solvents, or dilutants like, e.g. liquefied natural gas, which also acts as the propellant and reduces the amount of active ingredient atomized at the conventional high flow rates and/or reducing the viscosity of the active ingredient. However, in the practice of this invention, as in conventional apparatuses for atomizing a liquid product, the propellant itself may act as a solvent or dilutant for the liquid product when contained within the same compartment as the liquid product, i.e. when the propellant is liquefied natural gas, butane or chlorinated or fluorinated hydrocarbons.

In conventional systems, when liquid product is being dispensed, the effect of the propellant to act as a solvent or dilutant for the liquid product is significantly reduced as the propellant changes into its gaseous phase, being no longer available as a liquid solvent.

A known apparatus for atomizing liquid product is disclosed in US 5 921 439, using a nozzle to atomize a mixture of pressuring gas and liquid product. The liquid product and pressurizing gas form a mixture immediately before entering the atomizing nozzle but are delivered to the mixing compartment by separate tubes. In the storage compartments, the pressurizing gas exerts its pressure also on the liquid product, which is isolated from the pressurizing gas within a collapsible bag, surrounded by pressurizing gas.

From US 5 918 817 a two-fluid cleaning jet nozzle is known, which has an atomizing unit by which pressurized gas can atomize a liquid into droplets. This cleaning jet nozzle consists of two portions, namely a so-called atomizing tube and a cross-sectional area of $7 - 200 \text{ mm}^2$ into which the liquid and gas are introduced. This atomizing tube is provided with one exit port, which continues into an accelerating tube having a smaller diameter than the atomizing tube, namely $3 - 15 \text{ mm}^2$. As a result of the smaller cross-sectional area of the accelerating tube being fed from the atomizing tube which has a larger cross-sectional area, the velocity of the exiting fluid droplets is much higher than for conventional nozzles without a smaller diameter accelerating tube adjacent to the atomizing tube. In detail, this two-compartment jet nozzle provides almost double the exit velocity of atomized fluids at the same pressure of the propellant gas in comparison to the conventional jet nozzle, i.e. approaching the speed of sound at a supply pressure of gas about 3 bar. It becomes clear from the drawing, that the entrance port for the gas is always of a bigger cross section than the entrance port for liquid. This disclosure emphasizes the importance of a high velocity and a high volume to be obtained for the stream of liquid droplets in order to effectively remove contamination from the surface of silicon wafers.

Conventional aerosol spray systems typically produce flow rates of 0.5 to 3 g/s of product, where the product is a mixture of liquefied propellant gas, a dilutant or solvent and a small amount of active ingredient. In these systems, both the propellant gas and the dilutant or solvent are often volatile organic compounds such as butane and ethanol. These volatile organic

compounds are included to produce a spray with a "cool feel" as they quickly evaporate leaving behind just the active ingredient on the surface, e.g. the skin, or suspended in the air. Conventionally, a mixture of organic compounds is needed for adjusting the viscosity and solvency of the active ingredient, i.e. a liquid product without added volatile organic compounds. The volumetric ratio of gas (at atmospheric pressure) to active liquid product is typically between 2000 : 1 and 20.000 : 1. The propellant gas, the solvents and dilutants are released into the atmosphere, generating environmental problems.

For conventional atomizing valves a design is usually chosen which has an internal cavity volume arranged between afferent pathways for delivering liquid product and/or propellant and the exit, e.g. an atomizing nozzle of at least 100 mm³ and a total cavity volume including valve body, stem and actuator of between 100 and 300 mm³.

It is an object of the present invention to provide an apparatus for atomizing a liquid product using the pressure of a propellant which has a simple design and allows formation of small liquid droplets while requiring a significantly reduced amount of propellant gas in relation to the liquid product being atomized.

It is a further object of the present invention to provide an apparatus for atomizing a liquid product using pressure of a propellant which can effectively atomize a liquid product having a higher viscosity than e.g. water into small droplets while requiring a reduced amount of propellant.

It is a further object of the present invention to provide an apparatus for atomizing a liquid product, wherein the liquid product may be viscous, for example having a viscosity above that of e.g. water in order to avoid the use of a dilutant contained in the liquid product.

Furthermore, it is an object of the present invention to provide an apparatus for atomizing a liquid product, which liquid product may be viscous, using a comparatively low proportion of propellant to liquid product dispensed, while providing for a non-oscillating, i.e. stable stream of atomized liquid product at the exit port.

The present invention arrives at the above mentioned aims by providing an apparatus for atomizing a liquid product, using pressure of a gaseous propellant. The liquid product is atomized within a capillary tube. The apparatus is designed for a total flow rate from 0.5 grams per second to 0.01 grams per second, preferably from 0.3 grams per second to 0.05 grams per second through a single capillary tube. Further characteristic features of the apparatus and of the process using this apparatus are given in the attached claims.

The apparatus contains at least one capillary tube. One axial opening of the capillary tube is used for the discharge of the atomized liquid product, i.e. as an exit port. Also arranged on the capillary tube is at least one first entry port for entry of the liquid product which is distant from the exit port. At least one second entry port may be provided for entry of the propellant. By properly dimensioning the diameter of the capillary tube and the length or distance between the exit port and an adjacent entry port, either a first or a second entry port, the entering liquid product is atomized within the capillary tube by entering propellant. The liquid product is delivered to the first entry port by a pipe or tube, the propellant is delivered to the at least one second entry port by a separate pipe or tube. Depending on the type of storage container connected to this apparatus, a liquid product may be contained within the same container as the propellant or may be separated from the propellant. In case, both liquid product and propellant are contained within the same storage container, such as the conventional "dip tube" systems, some of the propellant may disperse or dissolve into the liquid product. However, the atomizing apparatus of the present invention can be used when essentially no propellant functions as a dilutant for the liquid product and the two components are separated and fed to their respective entry ports essentially separately. In case, liquid product and propellant are kept separated from each other, the propellant may still pressurize the liquid product, which may be contained for example in a collapsible bag or in a cylinder having a movable piston being pushed by the propellant, said cylinder being arranged within a canister containing the propellant. However, liquid product and propellant are physically separated from each other by phase. In case compressed gases like air or nitrogen are used as the propellant these compressed gases do not form a liquid phase at the pressure used. These gases may be in direct contact with the liquid product, although a small amount of dissolution of the gas phase into the liquid product may occur.

It is preferred that the first entry port is formed by the axial opening of the capillary tube opposite to the exit port and the at least one second entry port is arranged between the two axial openings.

As to proper dimensioning, a capillary tube as applicable for the present invention has an inner diameter of 0.1 mm to 1.0 mm, preferably 0.2 mm to 0.6 mm. An essential feature regarding the length of the capillary is that the length or distance between the exit port and the adjacent entry port, either a first or a second entry port, covers a range from of 5 mm to 100 mm, preferably 5 mm to 50 mm.

The diameters of the first and second entry ports are designed such that at normal atmospheric pressure a volumetric flow ratio of 1 : 50 to 1 : 5000, preferably from 1:100 to 1:300, of liquid product to propellant is adjusted. In general, the first entry port has a diameter from 0.1 mm to 2.0 mm, preferably from 0.2 mm to 1.0mm, more preferably from 0.3 mm to 1.0 mm, even more preferably from 0.4 mm to 0.7 mm. When used, the second entry port generally has a diameter from 0.1 mm to 0.7 mm, preferably from 0.15 mm to 0.50 mm, more preferably from 0.24 to 0.35 mm. The diameter of the first entry port may be formed by a flow restrictor in case the first entry port is the axial opening of the capillary tube. Such a flow restrictor may be formed by an insert into the capillary tube, decreasing its inner diameter.

Furthermore, such a flow restrictor, which decreases the inner diameter of the capillary tube may be inserted into the capillary tube between the exit port and the adjacent entry port.

As an alternative to separate first and second entry port for delivering liquid product and propellant to the capillary tube, respectively, an admixture of liquid product and propellant may be fed to the capillary tube, having just one entry port. For this embodiment, the same dimensions as described for the capillary tube apply. As the single entry port, for example the axial opening opposite to the exit port may be used.

This embodiment of a common afferent pathway for both liquid product and propellant to the capillary tube is applicable for instance in so called "dip-tube" systems, wherein the afferent pathway consists of a tubing reaching down into the liquid phase of admixed liquid product

together with liquefied propellant, which may be liquefied hydrocarbon, optionally chlorinated or fluorinated and connective cavities to the entry port of the capillary tube.

For the embodiments having one common afferent pathway for both liquid product and propellant, when using a propellant which forms a liquid phase at the pressure used, generating a liquid admixture of liquefied propellant with the liquid product itself, the afferent pathway has no need for a lateral opening, also referred to as vapour tap. However, when using a compressed gas as the propellant, which is phase-separated from the liquid phase, like e.g. compressed air or nitrogen, the dip-tube needs a lateral opening for admitting propellant into the afferent pathway in a section of the dip-tube which is not immersed in liquid product when the container is in the position where it is actuated to dispense liquid product.

Furthermore, in the embodiment of the "dip-tube", wherein the liquid propellant forms one phase with the liquid product itself, i.e. they are not separated by phase or physical barriers, the present invention achieves the atomization of liquid product within the capillary tube using only propellant forming a liquid phase with a liquid product, without the need for an additionally entry opening within the afferent tubing to allow entrance of additional gaseous propellant. This additional entry port, known from conventional atomizing apparatuses, also called vapour tap, allowing the additional entrance of gaseous propellant into the atomizing unit is not necessary for the present invention, when using the liquefied gases forming a liquid phase as the propellant. In order to reduce such high total flow rates of known dip-tube systems, they conventionally need a so-called vapour tap to allow inflow of additional propellant in its gaseous state, which reduces the flow rate of liquid mixture up the dip-tube. This reduction of the flow rate by additional gaseous propellant is used in conventional systems to reduce the amount of liquid product which is dispensed while maintaining a sufficiently high total flow rate which is necessary for a stable atomization.

Furthermore, it is an essential feature of the present invention that the low flow rate of propellant in relation to liquid product, when compared to conventional systems, allows to atomize the liquid product without oscillations of the flow at the exit port, i.e. without discontinuous bursts out of the exit port. In order to achieve a stable and continuous, i.e. non-oscillating flow of atomized liquid product out of the exit port, when using a comparatively low volumetric ratio of liquid product to propellant, and a comparatively low volumetric total

flow rate of propellant and liquid product it has been found that the internal dimensions of the afferent pathways to the capillary tube need to avoid internal spaces and cavities. In detail, the afferent tubings and pipes or the single pipe in the case of the dip-tube system, need to be connected to the capillary tube, including interposed valve mechanisms without internal cavities too large.

The internal cavity formed between afferent tubing and entry port into the capillary tube has a volume of below 50 mm^3 , preferably below 20 mm^3 , more preferably below 6 mm^3 and most preferably below 2 mm^3 .

With the low total volumetric flow rate the present invention achieves the same flow rate of liquid product (active ingredient) as the dilutants necessary in conventional systems can be omitted to a substantial degree. One reason is that the high viscosity of the liquid product is no longer an obstacle to atomization at low total flow rates. Another reason, more importantly, is that the present invention uses only comparatively low total flow rates of liquid product plus propellant.

It has been shown that conventional valve arrangements for atomizing apparatuses necessitate the use of flow rates of propellant and liquid product including any dilutants in the order of 0.5 g/s to 1.5 g/s in order to avoid unstable, i.e. oscillating flow. With lower total flow rates, the flow at the exit port becomes unstable and discontinuous, i.e. it oscillates. In order to reduce such high flow rates of propellant, dip-tube systems conventionally need a so-called vapour tap to allow inflow of additional propellant in its gaseous state.

In general, the combination of low total flow rate of propellant and liquid product and the low ratio of propellant to liquid product, which can be realized with the atomization apparatus according to the present invention, allows to dispense liquid product (active ingredient) at the same rate as conventional systems do, however, with less propellant and substantially less dilutants than conventionally necessary.

It has been found surprisingly that the volume of cavities containing the admixture of liquid product and propellant, which are created between the one or more afferent tubings and the actual atomizing capillary tube need to be controlled to be under a certain volume in order to

allow continuous and stable, i.e. non-oscillating flow to the exit port while still using low total flow rates and, additionally, low ratios of propellant to liquid product.

It is to be considered furthermore, that the diameter of the capillary tube atomizer affects the flow rate of the atomized liquid product inside the capillary by its inner diameter.

It has been found that the maximum cavity volume, defined as the void volume between the afferent pathway(s) for liquid product and/or propellant and the entrance port(s) to the capillary tube, can be determined experimentally by a person skilled in the art without undue experimentation to arrive at the dimensioning applicable in the present invention. As a guideline, the following considerations can be followed:

At a viscosity of 50 mPa • s (for example vegetable oil) the relationship can be calculated as:

$$\text{Maximum cavity volume allowed} = \text{Pressure}^{1.5} \exp [(d/R - 0.621)/0.2022]$$

At a viscosity of 1 mPa • s (for example water) this relationship changes to:

$$\text{Maximum cavity volume allowed} = \text{Pressure}^{1.5} \exp [(d/R - 0.4274)/0.1917],$$

where d is the capillary tube internal diameter in mm, and wherein R is the ratio of the diameters of the entry port for propellant to the entry port (which was a 40 mm long capillary tube) for liquid product (which may be defined by a restrictor inserted into the axial opening of the capillary tube), and wherein the internal diameter of the capillary tube is given in mm, the pressure is gauge and is given in bars and the maximum volume cavity allowed is calculated in mm³.

When a larger cavity volume than the above maximal cavity volume allowed is used, an unstable and/or oscillating flow is created when using the intended total flow rate.

From the above considerations, the skilled person is able to calculate and design sufficiently small cavity volumes even for values for viscosity and geometry being different from those

given above in order to arrive at a capillary tube atomizer which produces a continuous, i.e. non oscillating flow of atomized liquid product at low ratios of propellant to liquid product.

Furthermore, the above relations show that for a given system an increase in R or a decrease in the pressure applied can lead to unstable or oscillating flows.

The actual embodiments used as examples for calculating the above relations are given in the following examples. However, as a rule of thumb it is necessary to reduce the cavity volumes by a factor of 10 to 100 compared to the cavity volumes of conventional systems in order to arrive at a non-oscillating atomization of liquid product by comparatively lower ratios of propellant to liquid product. In embodiments according to the invention the cavity volume between the afferent pathway(s) and the entry port(s) to the capillary tube is between 0 and 20 mm³ and preferably below 10 mm³.

In the above calculations, when applied to a dip-tube system using just one afferent pathway between storage container and capillary tube with no additional opening within the afferent pathway for entrance of additional propellant, the ratio R becomes 1 and is to be replaced by the volumetric ratio of propellant to liquid product within the uniform mixture of liquid product and propellant.

In order to operate this atomizing apparatus, valves are used to open and to shut off the flow of the liquid product and/or propellant and/or the mixture of liquid product and propellant before the exit port. Therefore, a single on/off valve may be arranged on the capillary tube between the exit port and the adjacent entry port to completely block the capillary tube cross-section. In addition or as a separate embodiment, two valves may be arranged to separately block or regulate the flow of propellant to the second entry port and the flow of liquid product to the first entry port. These two valves may be actuated in parallel and simultaneously, however, it may also be provided for that the valve controlling the inflow of propellant into the second entry port admits propellant shortly before and after entry of liquid product in order to avoid liquid product accumulating in the capillary tube.

For the purposes of the specification, pressures given are defined as pressure gauge, i.e. the pressure above normal atmospheric pressure, unless otherwise indicated.

The propellant may be natural gas, like e.g. liquefied butane, propane or a halogenated or fluorinated hydrocarbon. However, an environmentally friendly propellant such as compressed air or nitrogen may be used as the propellant. In some cases, i.e. low flow rates of propellant necessary, even compressed carbon dioxide, compressed air or nitrogen may be used as the propellant.

When dimensioning the atomizer according to the invention, it is to be taken into account that the geometry will influence the flow rates of liquid product and propellant as well as the particle size of the droplets of liquid product produced. In detail, the particle size essentially depends on the ratio of diameters of first entry port to second entry port. Generally, the lower this ratio, the smaller the particles will be when both liquid product and propellant are under the same pressure.

The flow rate at the exit port is mainly a function of the inner diameter of capillary tube, e.g. a smaller inner diameter of the capillary tube will result in a lower flow rate at the same pressure for propellant and liquid product.

In accordance with the particle size being influenced by the ratio of cross-sections of the first entry port to the second entry port, the particle size is accordingly influenced by the volumetric ratio of liquid product to propellant. The lower the ratio of liquid product to propellant, the smaller the particles will be at the exit port.

Therefore, the following measures are to be taken to adjust the dimensions of the atomizer according to the invention:

If the particles produced at the exit port are too big, the ratio of liquid product to gas shall be decreased. In case of a separated storage of liquid product from propellant, like e.g. the liquid product contained within a collapsible bag compressed by the propellant, the ratio of the diameter of the first entry port to the diameter of the second entry port shall be decreased. In the case for the dip-tube arrangement, wherein propellant gas and liquid product are contained within the same canister, the volumetric ratio of liquid product to propellant shall be decreased.

In order to decrease the flow rate and the cross section of the exit port, the inner diameter of the capillary tube shall be decreased, or, alternatively, the ratio of liquid product to propellant shall be decreased.

In greater detail, an acceptable particle size initially combined with a flow rate too high at the exit port can be regulated by decreasing the inner diameter of the capillary tube or inserting flow restrictors into the capillary tube. Accordingly, an acceptable particle size initially combined with a flow rate too low at the exit port can be regulated by increasing the inner diameter, i.e. cross-section of the capillary tube.

In case the flow rate at the exit port is acceptable but the droplets produced are too large in size, the ratio of liquid product to propellant shall be decreased and the inner diameter of the capillary tube shall be increased. Accordingly, if the particles produced at the exit port are too small but the flow rate is acceptable, the ratio of liquid product to propellant shall be increased and the inner diameter of the capillary tube shall be decreased or flow restrictors shall be inserted.

The apparatus according to the invention is suitable for the atomization of liquid products having a dynamic viscosity from $0.3 \text{ mPa} \cdot \text{s}$ to $5000 \text{ mPa} \cdot \text{s}$.

As examples, the following design can be used for an atomizer according to the present invention of liquid product having the dynamic viscosity as indicated. In these examples, the liquid product was contained within a collapsible bag surrounded by propellant gas, both placed within a closed canister. Pressure of the propellant gas was approximately 3 bar gauge.

For the purposes of this specification, pressures given are defined as pressure gauge, i.e. the pressure above normal atmospheric pressure, unless otherwise indicated.

The first entry port was the axial opening of the capillary tube, the second entry port was arranged at a distance of 20 to 40 mm from the exit port.

Table 1

Example	dynamic viscosity [mPa • s]	diameter of first entry port [mm]	diameter of sec- ond entry port [mm]	capillary tube diameter [mm]
1	1 – 3	0.3 – 0.4	0.15 – 0.29	0.3 – 0.4
2	3 – 10	0.4 – 0.7	0.24 – 0.35	0.4 – 0.7
3	10 – 20	0.4 – 0.7	0.24 – 0.35	0.4 – 0.7
4	20 – 40	0.7 – 1.0	0.28 – 0.50	0.7 – 1.0

Examples 5 and 6 have been performed with a setup separating the liquid product from the propellant at a pressure of 2 bar and a distance of the exit port of the capillary tube from the adjacent second entry port of 40 mm, with the first entry port being the axial opening of the capillary tube opposite to the exit port.

Table 2

Example	dynamic viscosity [mPa • s]	diameter of first entry port [mm]	diameter of sec- ond entry port [mm]	capillary tube diameter [mm]	mass mean diameter of droplets [μm]
5	13	0.4	0.29	0.4	40
6	13	0.4	0.35	0.4	24

In a further embodiment of the present invention, the liquid product may be stored in a long tube of such a diameter that the flow of liquid into the first entry port is constant, if the valves are open. Such a tube may include a series of internal restrictions and, as the liquid is used up, the effective length of the tube is reduced. Therefore, less pressure is then required to create the desired flow of liquid and a decreasing pressure resulting from the compressed gas propellant being used up can be compensated by selecting tube length, tube diameter and re-

strictors. The droplet size was measured with a laser diffraction system, namely a Malvern particle size analyser.

Table 3

Example of dimensions for an atomizing apparatus with a storage compartment for liquid product separated from propellant, pressurizing the liquid product (bag-on-valve-type)

Atomized liquid product: oil (50 mPa • s)

Cavity Volume	Diameter of entry port for propellant	Diameter of entry port for liquid product	Pressure	Diameter of capillary tube	Flow rate
130 mm ³ (a)	0.5 mm	1.0 mm	2.7 bar	0.50 mm	0.60 g/s
Example 1 6 mm ³	0.5 mm	1.0 mm	2.7 barr	0.27 mm	0.15 g/s
Example 2 2 mm ³	0.5 mm	1.0 mm	2.7 bar	0.17 mm	0.06 g/s

(a) comparative example for a conventional spray can

Atomized liquid product: water (1 mPa • s)

Cavity Volume	Diameter of entry port for propellant	Diameter of entry port for liquid product (restrictor)	Pressure	Diameter of capillary tube	Flow rate
130 mm ³ (a)	0.27 mm	0.4 mm	2.7 bar	0.55 mm	0.60 g/s
Example 3 6 mm ³	0.27 mm	0.4 mm	2.7 bar	0.24 mm	0.12 g/s
Example 4 2 mm ³	0.27 mm	0.4 mm	2.7 bar	0.14 mm	0.04 g/s

(a) comparative example for a conventional spray can

A graphic representation of the results is given in figure 1.

Application examples

In the following, two embodiments of the apparatus according to the invention are compared for the same liquid product. The “bag-on-valve type” atomizing apparatus used a propellant, which is exchangeably compressed gas like air or nitrogen, which does not form a liquid phase at the pressures employed, as well as liquid natural gas. The propellant is contained within a container and has access to the capillary tube atomizer via a lateral entry port of the afferent pathway, whereas the liquid product is contained within a physically separated compartment like a collapsible bag or a cylinder with a movable piston, which compartment is connected to the afferent pathway, for example to one axial opening of an afferent tubing forming part of the afferent pathway.

The alternative embodiment, here termed “dip-tube”, employs one afferent pathway to the atomizing capillary tube, which afferent pathway does not have an additional entry port for e.g. gaseous propellant. In contrast, the afferent pathway only has one opening, for example the axial opening of an afferent tubing, which connects to the pathway leading to the capillary tube atomizer. Accordingly, a mixture of liquid product and liquid propellant enters into the afferent pathway, which mixture is not changed in respect of its ratio of propellant to liquid product by additional propellant entering the afferent pathway in its gaseous form.

Table 4

	Bag-On-Valve	Dip-Tube
Total flow rate of liquid product plus propellant	0.02 – 0.2 g/s (or higher)	0.05 – 0.3 g/s (or higher)
Viscosity of liquid product (active ingredients including solvents)	1 – 50 mPa • s	1 – 50 mPa • s
Propellant (volume)	20 – 80 %	20 – 80 %
Size of atomized particles	20 – 100 µm (a)	20 – 100 µm (a)
Spray angle	18° (16° - 20°)	18° (16° - 20°)

(a) mass mean diameter

Table 5

The following compositions for a hairspray may be used to produce exactly the same particle size and spray angle of atomized liquid product.

	Composition for conventional spray can	Composition for bag-on-valve or dip-tube system according to the invention
Resin (solid)	2 ml	2 ml
Propellant	30 ml	8 ml
Ethanol	50 ml	7 ml
Water	17 ml	3 ml
Total content	100 ml	20 ml
Concentration of resin	2 %	10 %
Total flow rate of system	1 g/s	0.2 g/s
Flow rate of resin	0.02 g/s	0.02 g/s
Reduction of propellant	n.a.	73 %
Reduction of ethanol	n.a.	86 %
Reduction of water	n.a.	82 %
Total content reduction	n.a.	80 %
Flow rate reduction	n.a.	80 %
Active reduction	n.a.	none

n.a. = non applicable, % = in relation to conventional spray can composition

When comparing the formulations for hairsprays which may be atomized using either a conventional spray can or the apparatus according to the invention for atomizing a liquid product, the same flow rate of active ingredients, which in this case is the solid resin, can be obtained while reducing the amount of propellant and dilutants when employing the atomizing apparatus according to the invention. In other words, the apparatus according to the invention for atomizing the liquid product allows to spray the same rate of active ingredients while using a lower total flow rate of liquid product plus propellant in combination with a reduced amount of propellant per amount of active ingredient.

The mass mean particle size is generally adjustable from 2 μm to 100 μm with the atomizing apparatus according to this invention.

The advantages of the apparatus for atomizing a liquid product according to the invention are that a very low total flow rate can be used to spray concentrated, e.g. viscous fluids, with a small amount of gaseous propellant. As examples for liquid fluids, air fresheners, insecticides, hair sprays, body sprays, perfumes and deodorants, colourant compositions, chemically active compositions, lubricants or fuel can be formed to droplets. As a high viscosity of the liquid product is no further an obstacle to atomizing, at such low total flow rates the apparatus for atomizing according to this invention nearly eliminates the need for volatile organic compounds such as alcohols, butane or dimethylether as dilutants to be included into the liquid product for reducing its viscosity.

Although the formation of small droplets from the liquid product is achieved within the capillary tube which is fed by the liquid product and propellant, an additional small nozzle may be provided at the exit port for further decreasing the droplet size.

For regulating and actuating the apparatus according to the invention, valves can be located at several positions. In one embodiment, a central valve can be arranged on the capillary tube between the exit port and the adjacent entry port in order to block further movement of propellant and atomized liquid product towards the exit port. However, this embodiment is disadvantageous in respect of possible mixing of propellant and liquid product via the connecting portion of the capillary tube, where liquid product is separately stored from the propellant, like for example in a collapsible bag arranged within the propellant contained in a canister.

As a further embodiment, two separate valves can be used to block the pipe or tubings delivering liquid product and propellant to the first and second entry ports, respectively. These two valves can be actuated simultaneously or in such a manner that the valve controlling the second entry port allows inflow of propellant before, during and after liquid product is admitted into the capillary tube.

Furthermore, valves may be used which meter the amount of liquid and/or propellant so that for each actuation of the valves, an adjustable amount is dispensed.

When employing the apparatus according to the invention for atomizing a liquid product, the liquid product, i.e. active ingredient can be dispensed with only a small amount or no dilutant. Therefore, the liquid product is highly concentrated and very small flow rates can be achieved in comparison to conventional systems. As a consequence, the liquid product can reach for example skin without a large amount of dilutants like volatile organic compounds, resulting in a dry feel of the atomized liquid product as only little or no energy is necessary for the evaporation of volatile organic compounds. When using the atomizing apparatus according to the present invention, the flow rate of active ingredient, as defined, with only small amounts of dilutants necessary for dissolving or dispersing the actual active ingredient, the flow rate of active ingredient can remain at the same level as in conventional systems, however, using a greatly reduced total flow rate of propellant and the active ingredient combined.

The present invention uses pressures for the gaseous propellant from 2 bar to 5 bar (200 kPa to 500 kPa), preferably 2 bar to 4 bar and even more preferably 2 bar to 3 bar.

The total flow rate within the capillary tube within which atomization of liquid product takes place is restricted to the range specified above. In order to scale up the total flow rate of an apparatus for atomizing liquid product within a capillary tube a plurality of capillary tubes may be used which are arranged in a bundle, a row or in another way. Every capillary tube of such plurality of capillary tubes may be supplied with liquid product to be atomized and propellant taken from the same source respectively. A few capillary tubes for atomizing liquid product may be supplied with several different liquid products and the same propellant or several propellants taken from the same source or different sources. In this case the liquid products come into contact with each other after the single liquid product has been atomized. The liquid product to be atomized and the propellant may be taken out of containers having relatively small volumes which are combined with preferably one or a few capillary tubes. This arrangement may result in a handheld unit.

Furthermore, the liquid product to be atomized and the propellant may be taken out of containers having relatively large volumes or may be taken out of pipelines. These pipelines are

preferably connected to a plurality of capillary tubes. In this case a continuous or quasi continuous operation of the atomizer is possible. This arrangement may result in a stationary or mobile unit for continuous or quasi continuous atomization of liquid product. The total flow rate of such a unit is appreciably greater than the total flow rate through only one of the single capillary tubes.

The present invention will now be described in greater detail with reference to the embodiments of the invention described in the figures. Identical reference numbers refer to respective parts.

Figure 1 is a graphical representation of the experimental results described in table 3.

For clarity of demonstration the following figures 2 to 19 show embodiments of the apparatus according to the invention for atomizing liquid product using pressure of a gaseous propellant wherein only a single capillary tube is used within which the liquid product is atomized. Embodiments using a plurality of capillary tubes within each of which atomization of liquid product takes place are not shown.

Figure 2 schematically shows a first embodiment of the apparatus according to the invention, wherein a canister 1 contains a propellant 2. A flexible bag 3, arranged within the canister 1, contains the liquid product 7 and is pressured by the propellant 2. The flexible bag 3 is connected to the capillary tube 4 via valve 8, which in this case also allows the entry of propellant into the capillary tube 4. The capillary tube 4 is open to the environment at its exit port 5.

In figure 3, showing a further embodiment of the apparatus according to figure 2, liquefied gas 6 is contained within the canister 1 from which a propellant 2 is formed.

Figure 4 shows a section of the capillary tube used for atomizing the liquid product according to the invention. The capillary tube 4 has an inner passageway 12, which is open to the environment at the exit port 5. Entry ports 13, 14, used as first and second entry ports, respectively or vice versa allow the entry of liquid product and propellant into passageway 12. At entry port 13, a flow restrictor 11 is shown. When the on/off valve 9 is open, liquid enters to the

entry port 13 within the restrictor 11 and passageway 12. The gaseous propellant enters at entry port 14. The pressure difference towards exit port 5 drives liquid product and gaseous propellant through the capillary tube, which causes the atomization of the liquid product inside the capillary tube.

In case a canister is used to store liquid product and propellant, both are at the same pressure.

Figure 5 shows a capillary tube 4, wherein common entry port 15 is used for allowing the entrance of propellant and liquid product in admixture.

Figures 6 to 9 show different arrangements of flow restrictors 11 and valve 9 to control the flow rates of propellant, liquid product and their admixture, respectively. Flow restrictors 11 and valves 9 can be arranged at different positions within the pathway for liquid product, propellant and their admixture, before or after the entry into the capillary tube 4.

Figures 10 and 11 show a canister 1 with the attached atomizing apparatus according to the present invention. A flexible bag 3 is connected to the capillary tube 4 via a bore 10 as an afferent pathway allowing the entry of liquid product from the flexible bag 3 into the first entry port 13, which is guarded by valve arrangement 16. Propellant is admitted to the second entry port 14 via bore 18 as a second afferent pathway, allowing entry of propellant into the capillary tube via the second entry port 14, which is guarded by the valve arrangement 17. When pushing (arrow) the capillary tube 4 axially into canister 1, valve arrangements 17 and 16 are opened for dispensing liquid product, being atomized within the capillary tube and being propelled by propellant through exit port 5. The valve arrangements 16 and 17 may comprise an annular seal like an O-ring. Figure 10 shows the apparatus in the inactive state, Figure 11 shows the same apparatus in the active state. Note that this embodiment avoids any cavity for the admixture of product and propellant.

Figure 12 shows a similar arrangement to that of figure 10, but using a capillary tube 4 which is closed at its axial end opposite to the exit port 5 and has one common lateral entry port 15. The gaseous propellant 2 mixes with liquid product 7 after passing bore 18. There is no separate valve arrangement for regulating the inflow of liquid product into the capillary tube 4,

however, valve arrangement 17 regulates the inflow of the mixture of gaseous propellant and liquid product into capillary tube 4 via annular cavity 19.

Figures 13 to 19 demonstrate embodiments of the atomizing apparatus, wherein cavity 19, arranged between afferent pathway 20 and the capillary tube 4 is dimensioned to have small volume.

In figure 13, applicable for example in a dip-tube system using liquefied gas as the propellant, a cover or lid 21 can be seen for fastening to a gas-tight canister with a sealing ring 22. Housing 23 for a valve is threaded into a threaded bore of cover 21 and sealed by a gasket 24 to cover 21. The gasket 24 engages an annular groove of stem 25 extending outwardly through a bore of cover 21 and inwardly into the inner space of housing 23. Coil spring 26 biases the stem 25 upwardly against gasket 24. The stem 25 contains the capillary tube 4, having a small inner diameter. At the lower end of capillary tube 4, a transverse bore 27 in stem 25 is provided, which is closed by gasket 24 when coil spring 26 is in its extended state. The transverse bore 27 acts as common entry port 15, however, a transverse second bore 27 may be provided. The afferent tubing 20 is formed by a pipe which extends through an eccentric bore of the housing 23 into cavity 19.

This embodiment is suitable for so-called dip-tube systems, wherein the propellant is for example liquefied natural gas, optionally chlorinated or fluorinated, which forms a liquid mixture with the liquid product and is guided as one mixture through the afferent tubing 20. In order to keep the volume of cavity 19 small, it is preferred that there is little or no connection to the space wherein coil spring 26 is arranged, i.e. the inner part of stem 25 essentially seals the bore of housing 23, wherein coil spring 26 is contained.

In figure 14 an embodiment of the invention is shown with a cover 21 which can be fastened to a conventional metal can (not shown) which is used for conventional spray packs. The housing 23 is fixed within the dome of the housing 23 and supports the afferent tubing 20. The upper part of the housing 23 contains a coil spring 26, which urges the lower part of stem 25 against sealing gasket 24, which in turn engages an annular groove of stem 25. Gasket 24 seals lateral bore 18 in the upper portion of the stem, which is connected with an elongated passage, which axially continues into capillary tube 4. The lower portion of the housing 23 has an afferent bore 28, which is connected to cavity 19, separated from the bore 18 by the

gasket 24. Afferent bore 28, being positioned higher than the opening of afferent tubing 20 as suitable for admitting gaseous propellant into cavity 19, whereas afferent tubing 20 allows the entry of liquid product into the room occupied by coil spring 26 and, through an intermediate space between the bore of housing 23 and stem 25 into cavity 19. When stem 25 is pushed axially to compress coil spring 26, gasket 24 is no longer positioned to seal bore 18, now admitting the mixture of gaseous propellant and liquid product, formed in cavity 19, into capillary tube 4. Such an embodiment is suitable for so-called bag-on-valve type spray cans, wherein the liquid product is physically separated from the surrounding propellant by for example a collapsible bag or a tube with movable piston, allowing pressurization of liquid product by the pressurizing propellant. The liquid product is only admitted into afferent tubing 20, whereas the gaseous propellant only enters afferent bore 28. However, such an embodiment may also be used in cases, where liquid product and propellant are not separated by a physical barrier but by phase-separation, for instance when the propellant is compressed air or compressed nitrogen, which do not form a substantial liquid phase and dissolves into the liquid product only to a small amount.

In figure 15, a separate arrangement from figure 14 is shown, in figure 14 both liquid product and propellant are admitted via separate afferent tubings to cavity 19, where they mix and enter the capillary tube 4 when stem 25 is pushed so that gasket 24 opens the bore 18. In figure 15, afferent bore 28, admitting propellant, is formed as an annular space between afferent tubing 20 and housing 23. Afferent tubing 20 admits liquid product via connecting bores 36 and 37 to cavity 19. The sealing 29 prevents removal of afferent tubing 20 and admixture of propellant and liquid product prior to their entering cavity 19. This embodiment may be used for the same applications as that of figure 14.

As an alternative embodiment, figure 16 shows afferent tubing 20 for liquid product and bore 28 for gaseous propellant, respectively, before they are admitted to cavity 19. Cavity 19 opens into a lateral bore 18 when stem 25 is pushed axially for removal from gasket 24 and further connects to capillary tube 4. This embodiment may be used for the same applications as that of figure 14.

In figure 17, liquid product is admitted by afferent tubing 20, which allows entry into the space occupied by coil spring 26 within housing 23. Gasket 30 seals the first entry port 13 and

gasket 24 seals the second entry port 14, when coil spring 26 urges stem 25 in its extended state. Afferent bore 28 connects to an annular space between housing 23 and stem 25 via lateral bore 18. When pressing stem 25, second entry port 14 is opened by removal from gasket 24, whereas first entry port 13 is opened by removal from gasket 30 to allow gaseous propellant and liquid product, respectively, to enter into space 31, which connects to the capillary tube 4. In an upright position, however, space 31 is filled with liquid product and a cavity 19 forms at the top end of space 31 adjacent capillary 4. This embodiment is suitable for the same purposes as the embodiment of figure 14.

In figure 18, afferent tubing 20 conducts liquid product into a chamber 33, separated from chamber 34 by interposed flexible partition wall 32. The flexible partition wall 32 is received in annular grooves of stem 25 and housing 23, respectively, biasing stem 25 against cover 21. Chamber 33 may connect to lateral bore 35 when gasket 24 is bent by depressing stem 25. Gaseous propellant is admitted via lateral bore 28 into chamber 34, which connects to bore 18 when gasket 24 is bent by the stem 25 being depressed. Within space 31, corresponding to cavity 19, liquid product and gaseous propellant are mixed before entering the capillary tube 4, thus avoiding substantial cavities within the afferent pathway of the mixture of liquid product and propellant before capillary tube 4. The embodiment of figure 18 may be used for the same purposes as the embodiment according to figure 14.

Figure 19 shows a "bag on valve" arrangement of the apparatus according to the invention. The gaseous propellant enters through afferent bore 28. The liquid product is stored in flexible bag 3 and enters through afferent tubing 20 discharging the liquid product into cavity 19 where it is mixed with the gaseous propellant. The mixture enters the capillary tube 4 via common entry port 15.

List of reference numbers

1	canister	20	afferent tubing
2	propellant	21	cover
3	flexible bag	22	sealing ring
4	capillary tube	23	housing
5	exit port of capillary tube	24	gasket
6	liquefied gas	25	stem
7	liquid product	26	coil spring
8	valve	27	transverse bore
9	valve	28	afferent bore
10	bore	29	sealing
11	flow restrictor	30	gasket
12	inner passageway	31	space
13	first entry port	32	flexible partition wall
14	second entry port	33	chamber
15	common entry port	34	chamber
16	valve arrangement	35	lateral bore
17	valve arrangement	36	connecting bore
18	bore	37	connecting bore
19	cavity		

Claims

1. An apparatus for atomizing a liquid product using pressure of a gaseous propellant, the liquid product being atomized within a capillary tube, the apparatus being designed for a total flow rate from 0.5 to 0.01 grams per second through a single capillary tube, comprising
 - at least one capillary tube with one exit port in its axial direction for discharge of liquid product and gaseous propellant, and
 - at least one entry port into the capillary tube distant from the exit port, and
 - at least one afferent pathway for delivery of liquid product and propellant in admixture or separately to the at least one entry port, the afferent pathway comprising afferent tubing(s) for liquid product and/or propellant where any connective cavity to the at least one entry port carrying the liquid product and propellant in admixture has an internal cavity volume of below 50 mm³, and
 - at least one valve for operating the apparatus.
2. An apparatus of claim 1, being designed for a total flow rate from 0.3 grams per second to 0.05 grams per second through a single capillary tube.
3. An apparatus according to claim 1 for atomizing a liquid product using pressure of a gaseous propellant, characterized in that
 - the at least one capillary tube having one exit port in its axial direction for discharge of atomized liquid product and gaseous propellant, and
 - the at least one capillary tube having at least one first entry port for entry of the liquid product being arranged distant from the exit port and having at least one second entry port being arranged distant from the exit port for entry of the propellant, and
 - the at least one capillary tube having a sufficient internal diameter and length between the exit port and the at least second entry port to allow atomization of entering liquid product by entering propellant, and
 - the at least one first entry port and the at least one second entry port having diameters to allow entrance of liquid product and gaseous propellant in a volumetric flow ratio of from 1:50 to 1:5000, preferably from 1:100 to 1:300, of liquid product to gaseous propellant.

4. An apparatus according to claim 1 for atomizing a liquid product using pressure of a gaseous propellant, characterized in that
 - the at least one capillary tube having one exit port in its axial direction for discharge of atomized liquid product and gaseous propellant, and
 - the at least one first entry port for entry of the liquid product being arranged distant from the exit port, and
 - the at least one second entry port for entry of the gaseous propellant being arranged between the first entry port and the exit port, and
 - the at least one capillary tube having a sufficient internal diameter and length between the exit port and the at least second entry port to allow atomization of entering liquid product by entering propellant, and
 - the at least one first entry port and the at least one second entry port having diameters to allow entrance of liquid product and gaseous propellant in a volumetric flow ratio of from 1:50 to 1:5000, preferably from 1:100 to 1:300, of liquid product to gaseous propellant.
5. An apparatus according to claim 1 for atomizing a liquid product using pressure of a gaseous propellant, characterized in that
 - the at least one capillary tube having one exit port in its axial direction for discharge of atomized liquid product and gaseous propellant, and
 - the at least one first entry port for entry of the liquid product being arranged distant from the exit port, the first entry port being arranged axially at the end of the capillary tube opposite to the exit port, and
 - the at least one second entry port for entry of the gaseous propellant being arranged between the first entry port and the exit port, and
 - the at least one capillary tube having a sufficient internal diameter and length between the exit port and the at least second entry port to allow atomization of entering liquid product by entering propellant, and
 - the at least one first entry port and the at least one second entry port having diameters to allow entrance of liquid product and gaseous propellant in a volumetric flow ratio of from 1:50 to 1:5000, preferably from 1:100 to 1:300, of liquid product to gaseous propellant.
6. An apparatus according to claim 1 for atomizing a liquid product using pressure of a gaseous propellant, characterized in that

- the at least one capillary tube having one exit port in its axial direction for discharge of atomized liquid product and gaseous propellant, and
 - the at least one entry port for entry of the liquid product and of the propellant being arranged distant from the exit port, and
 - the at least one capillary tube having a sufficient internal diameter and length between the exit port and the at least one entry port to allow atomization of entering liquid product by entering propellant, and
 - the at least one entry port having a diameter to allow entrance of liquid product and finally gaseous propellant in a volumetric flow ratio of from 1:50 to 1:5000, preferably from 1:100 to 1:300, of liquid product to finally gaseous propellant, and
 - wherein the liquid product is pressurized by gaseous propellant and wherein the liquid product is mixed with liquefied propellant within a compartment which is connected to one afferent pathway, wherein the afferent pathway is disposed for delivering both the liquid product and the liquefied propellant to the at least one entry port without a lateral opening in the afferent pathway.
7. An apparatus according to claim 1 for atomizing a liquid product using pressure of a propellant, which under the pressure applied forms a gaseous phase only, essentially separated from the liquid product, characterized in that
- the at least one capillary tube having one exit port in its axial direction for discharge of atomized liquid product and gaseous propellant, and
 - the at least one entry port for entry of the liquid product and of the gaseous propellant being arranged at the end of the at least one capillary tube opposite to the exit port, and
 - the at least one capillary tube having a sufficient internal diameter and length between the exit port and the at least one entry port to allow atomization of entering liquid product by entering propellant, and
 - the at least one entry port having a diameter to allow entrance of liquid product and gaseous propellant in a volumetric flow ratio of from 1:50 to 1:5000, preferably from 1:100 to 1:300, of liquid product to gaseous propellant, and
 - wherein the liquid product is pressurized by the gaseous propellant contained within the same container, wherein the afferent pathway is disposed for delivering the liquid product to the entry port, and

- wherein the afferent pathway has a lateral opening for entry of gaseous propellant separate from liquid product and wherein the afferent pathway between the lateral opening and the entry port has only small internal cavities to allow for a non-oscillating flow of liquid product and gaseous propellant in the capillary tube.
8. An apparatus according to claim 6, wherein the afferent pathway provides an afferent tubing whose axial opening is arranged within the vicinity of that end of the container opposite to the capillary tube and has a large inner diameter to allow the atomization of liquid product when the container is inverted such that the axial opening of the afferent tubing is not immersed in the mixture of liquid product and propellant.
 9. Apparatus to one of the preceding claims, wherein distance between the exit port and the at least one entry port for entry of the gaseous propellant is from 5 mm to 100 mm, preferably from 5mm to 50 mm.
 10. Apparatus according to one of the preceding claims, wherein the first entry port has a diameter of from 0.1 mm to 1.0 mm, preferably from 0.2 mm to 0.6 mm.
 11. An apparatus according to one of the preceding claims, wherein the diameter of the capillary tube is locally reduced by at least one flow restrictor, inserted either between the exit port and the adjacent entry port and/or between the first and second entry ports.
 12. An apparatus according to one of the preceding claims, wherein the internal cavity formed between afferent tubing(s) and the entry port(s) of the capillary tube has a volume of below 20 mm^3 , preferably below 6 mm^3 and most preferably below 2 mm^3 .
 13. An apparatus according to one of the preceding claims, wherein one valve is arranged on the capillary tube between the exit port and the adjacent entry port.
 14. An apparatus according to one of the preceding claims, wherein valves are arranged each at the first and second entry ports.

15. An apparatus according to one of the preceding claims, wherein a nozzle is provided at the exit port, e.g. a swirl chamber nozzle.
16. An apparatus according to one of the preceding claims, wherein the capillary tube is bent.
17. An apparatus according to one of the preceding claims, wherein the capillary tube is coiled.
18. An apparatus according to one of the preceding claims, wherein a stopper is provided within the afferent pathway for blocking it and then opening an alternative pathway to allow the atomization of liquid product when the apparatus is turned to a position upside down, by which the atomizing capillary tube points downwards.
19. An apparatus according to one of the preceding claims, wherein a filter mesh or membrane, which is permeable to gas but impermeable to liquids is arranged at the entry port(s) for propellant within the afferent pathway.
20. An apparatus according to claim 1 comprising
 - a plurality of capillary tubes with one exit port each in the axial direction for discharge of liquid product and gaseous propellant, every single capillary tube being designed as described in the preceding claims, and
 - at least one entry port into every single capillary tube distant from the exit port of every single capillary tube, and
 - an internal cavity formed between afferent tubing to every single capillary tube and at least one entry port into every single capillary tube, the internal cavity having a volume of below 50 mm³ for every single internal cavity, and
 - at least one valve for operating the apparatus.
21. An apparatus according to claim 20 comprising
 - a plurality of capillary tubes arranged parallel to each other.
22. An apparatus according to claim 20 comprising
 - a plurality of capillary tubes arranged in a bundle of capillary tubes.

23. An apparatus according to claim 20 comprising
- a plurality of capillary tubes which are inclined in respect to each other.
24. An apparatus according to claim 20 comprising
- a plurality of capillary tubes wherein every single capillary tube is connected to the same source of liquid product and to the same source of propellant.
25. An apparatus according to claim 20 comprising
- a plurality of capillary tubes wherein the capillary tubes are connected in groups to containers containing different liquid products and different propellants.
26. An apparatus according to claim 20 comprising
- a plurality of capillary tubes wherein the capillary tubes are connected to pipelines for the supply of liquid product and of propellant.
27. Process for dispensing a liquid product using an apparatus according to one of the preceding claims.
28. Process according to claim 27, wherein the flow rate of the liquid product is from 0.01 grams per second to 0.4 grams per second through a single capillary tube.
29. Process according to claim 7 and 28, wherein the liquid product is one of the group of cosmetic preparations, paint compositions, chemically active compositions, foaming compositions, lubricants or fuels.
30. Process according to one of claims 27 to 29, wherein the propellant is
- one of the group of compressed air, nitrogen, carbon dioxide, hydrocarbon, helium, neon, or
 - one of the group of liquefied gases free of halogens, propane, butane, pentane, ether, dimethylether, diethylether, or
 - one of the group of halogenated liquefied gases, or
 - a mixture of gases, or
 - a mixture of liquefied gases.

EXPERIMENTAL RESULTS
(definitions correspond to description of figure 1)

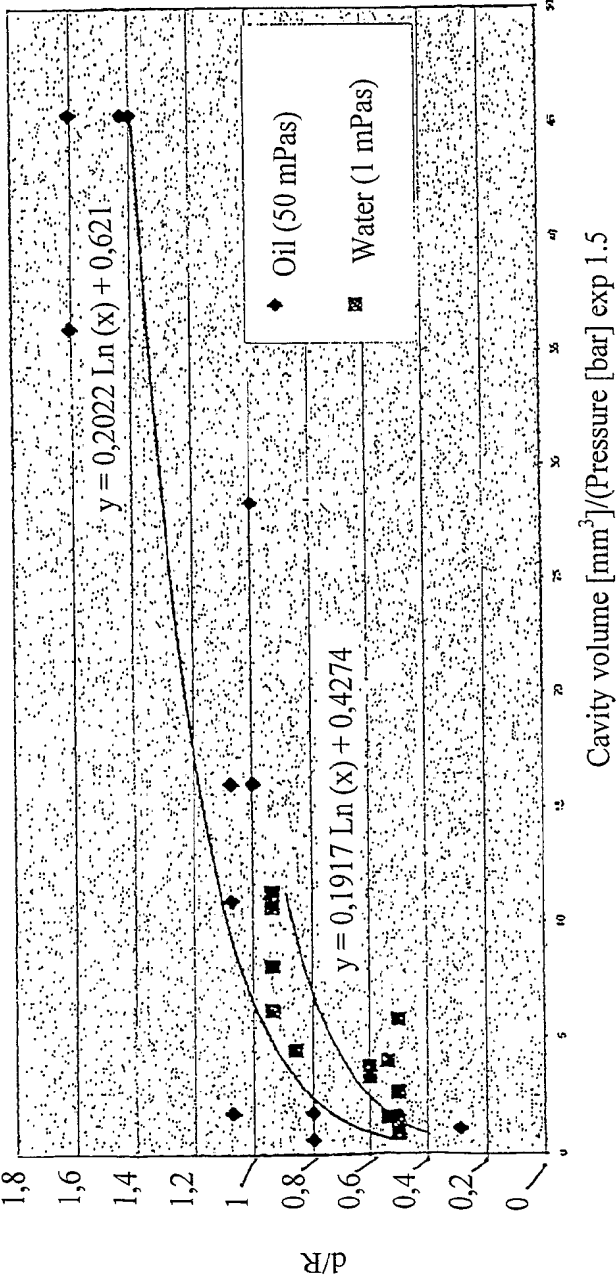
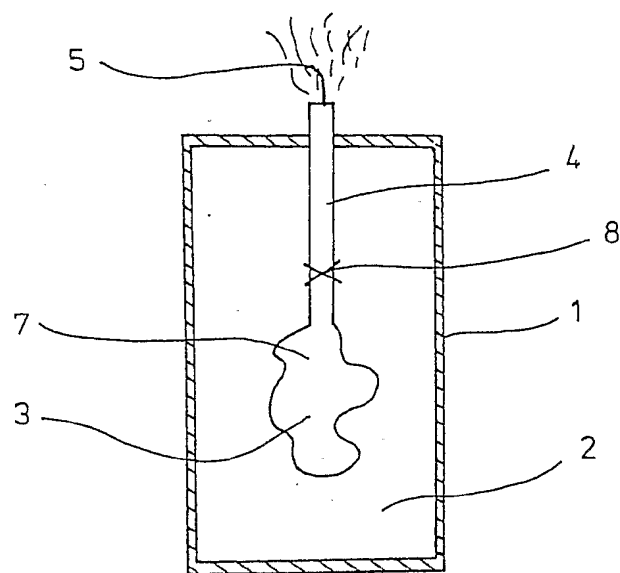
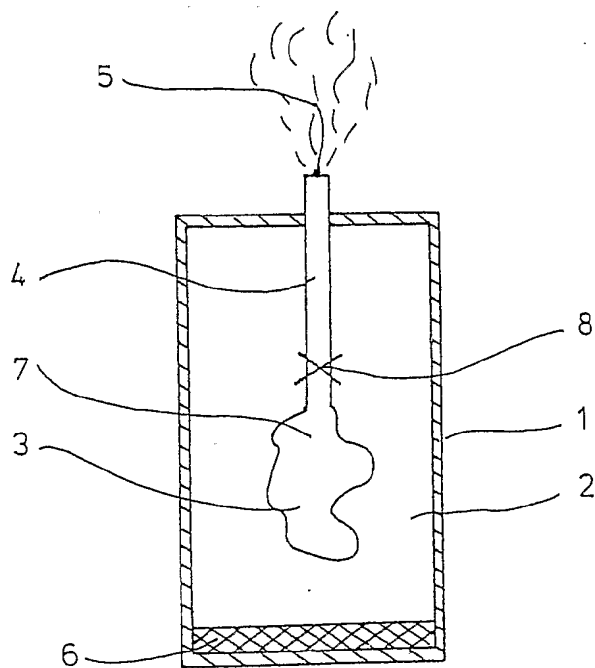
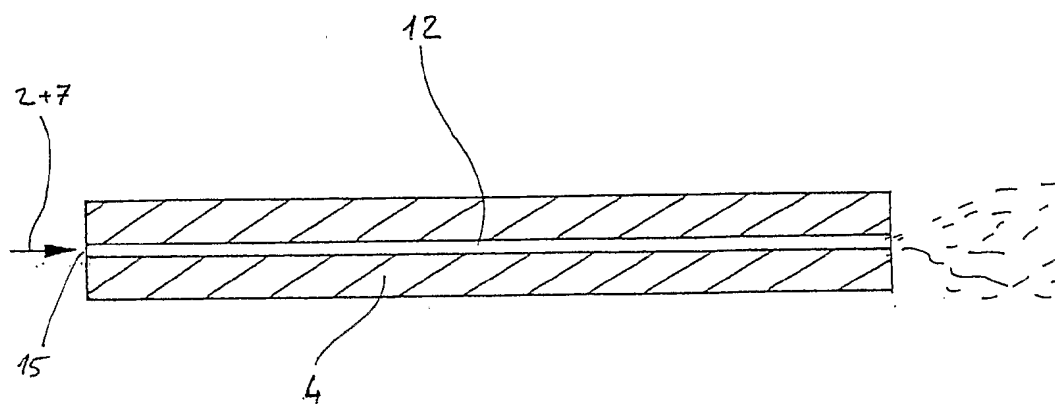
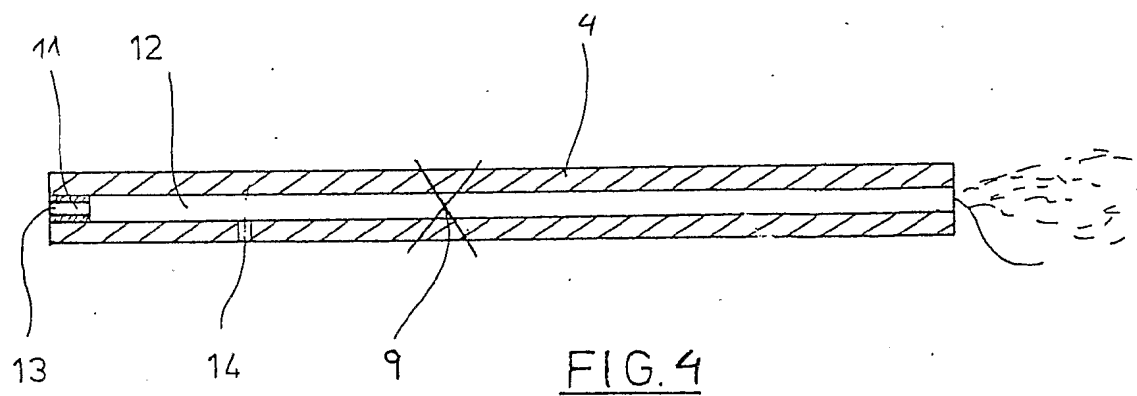


Fig 1

FIG. 2FIG. 3

FIG. 5

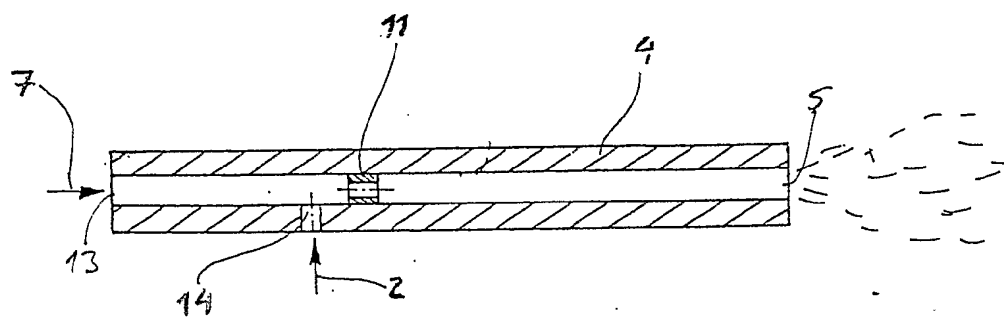


FIG. 6

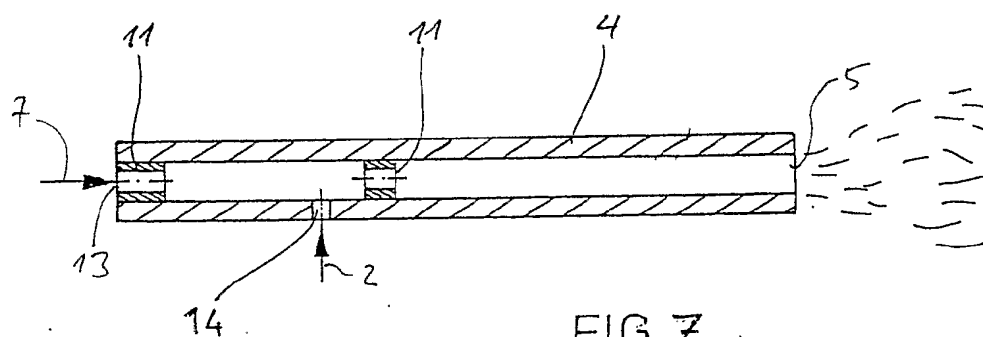


FIG. 7

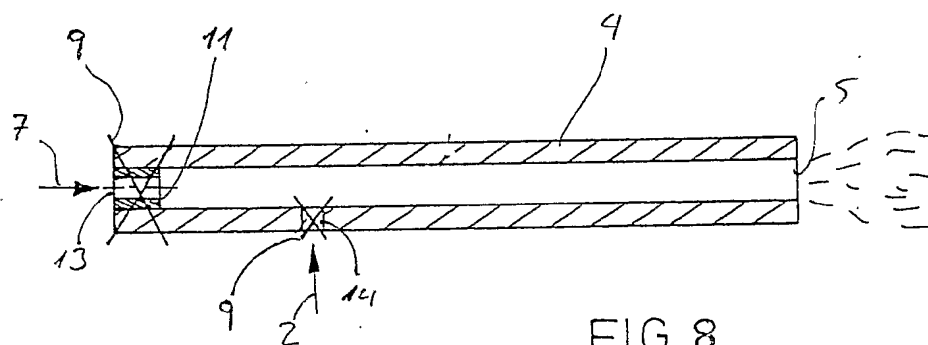
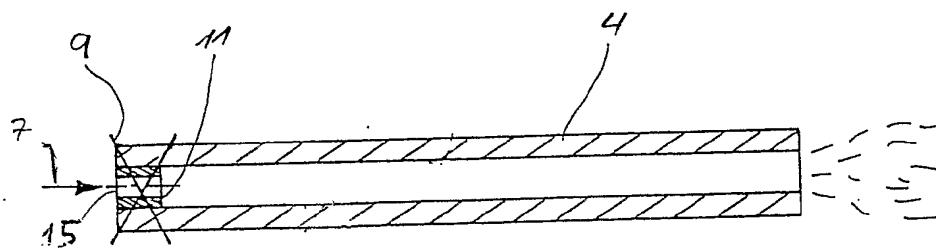


FIG. 8

FIG. 9

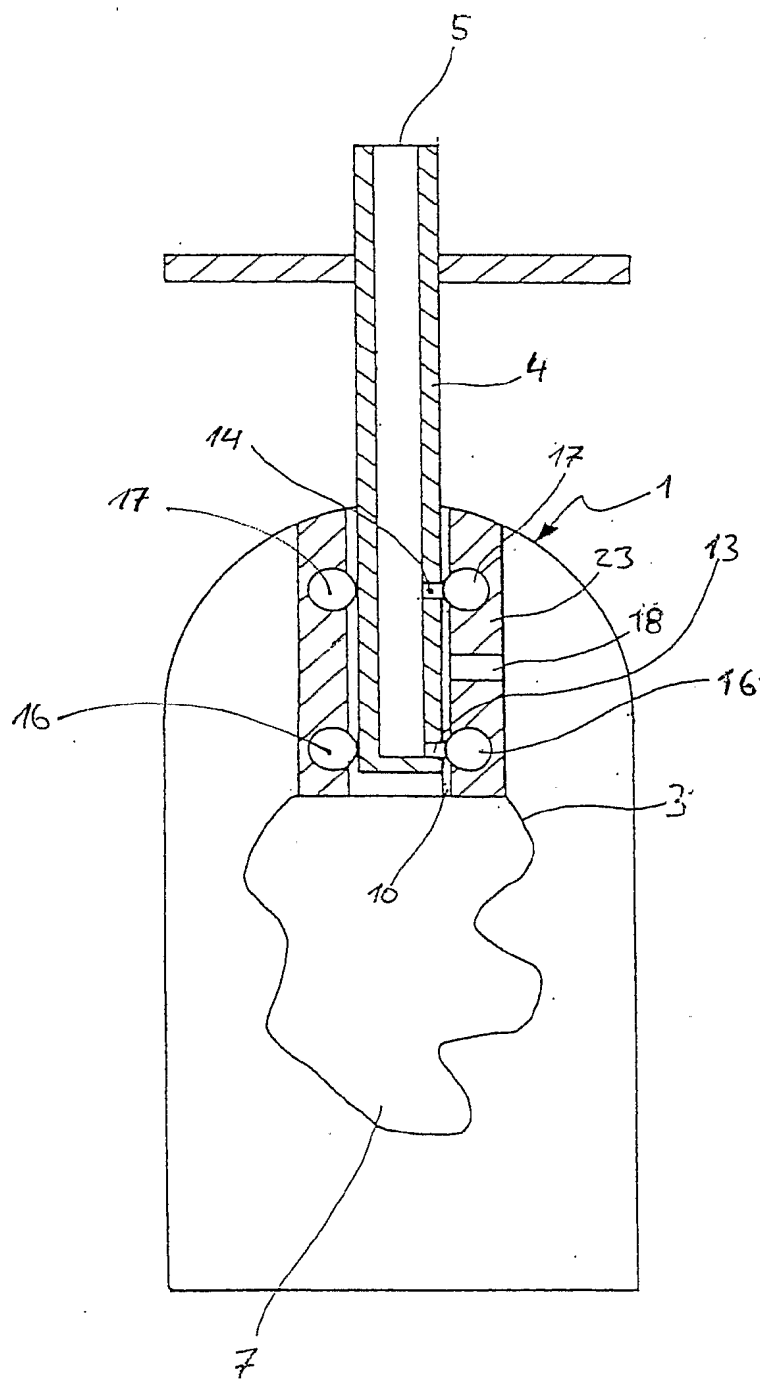


FIG.10

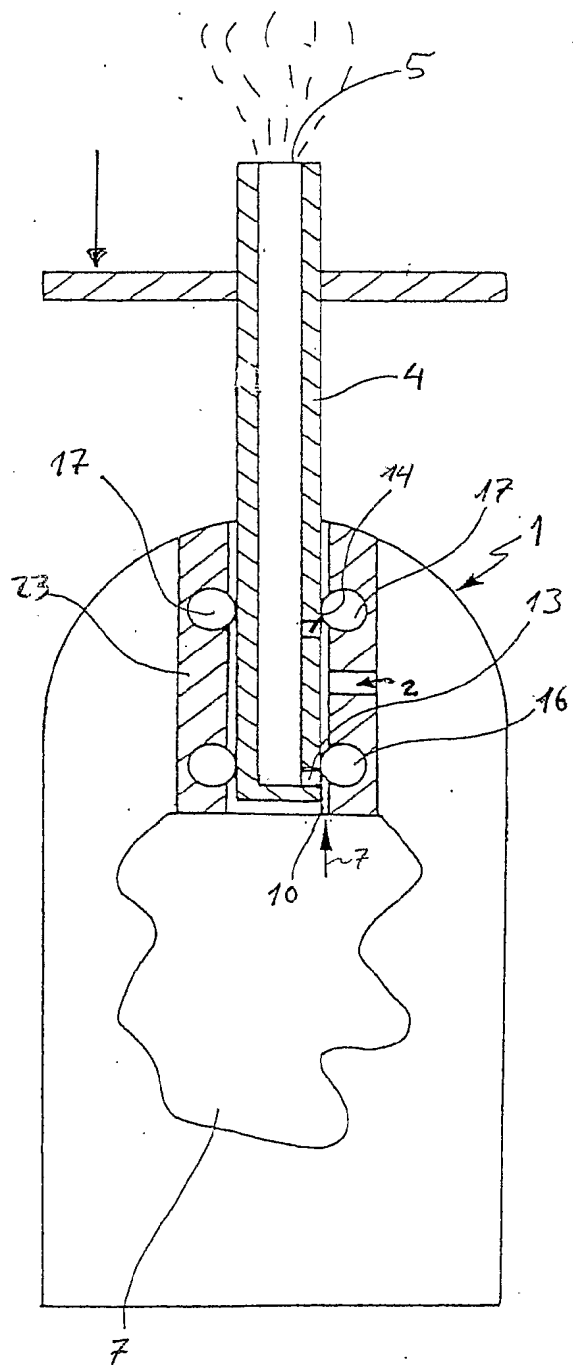
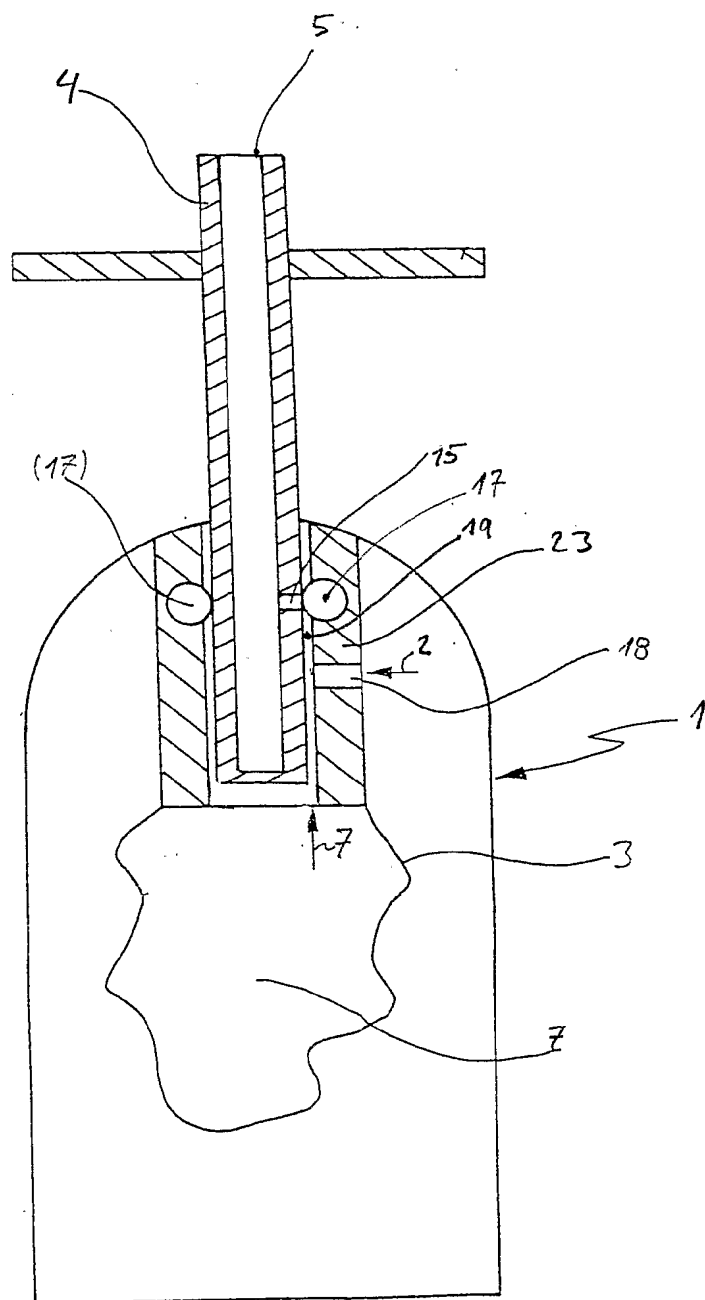


FIG.11

FIG. 12

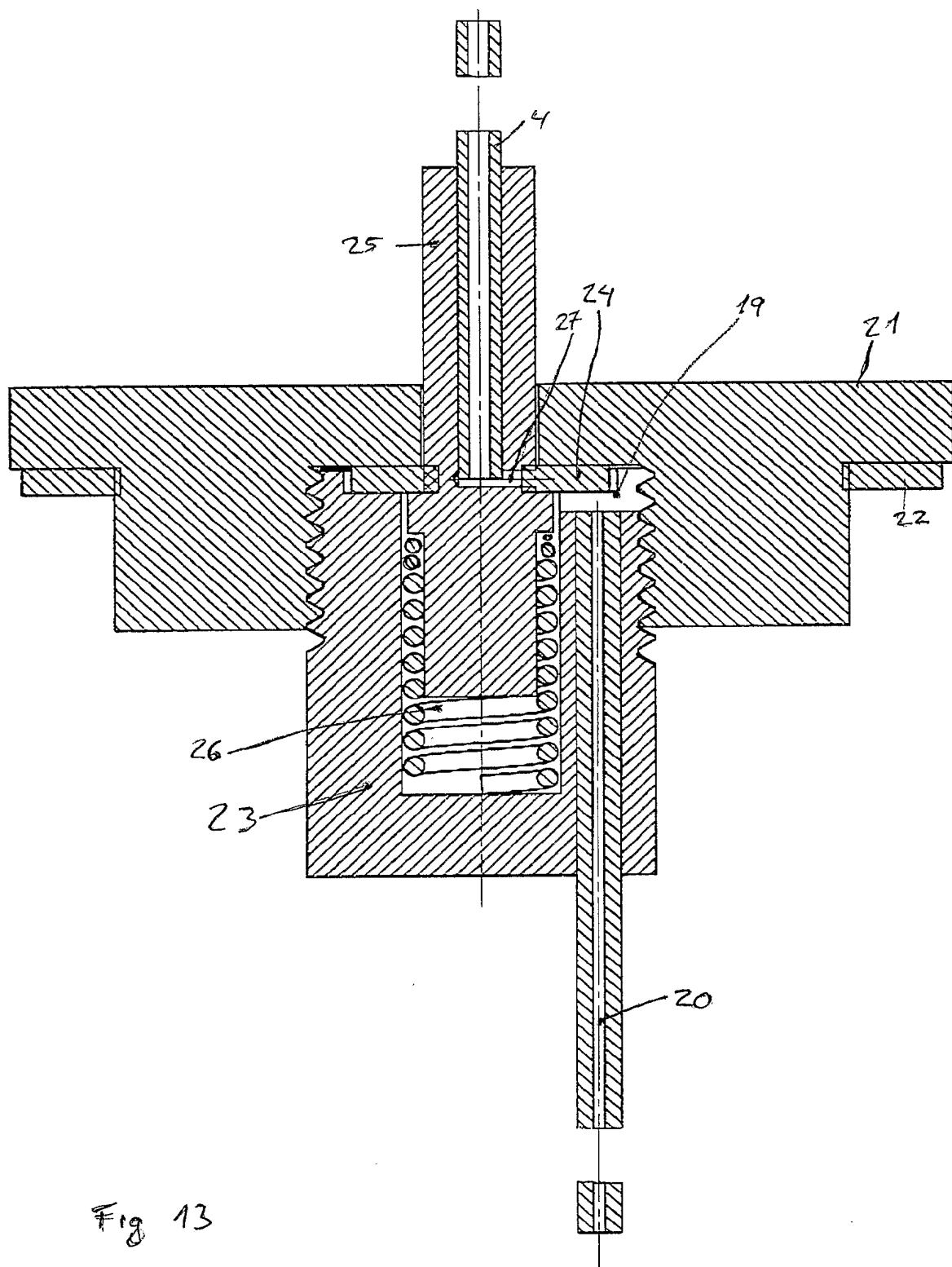
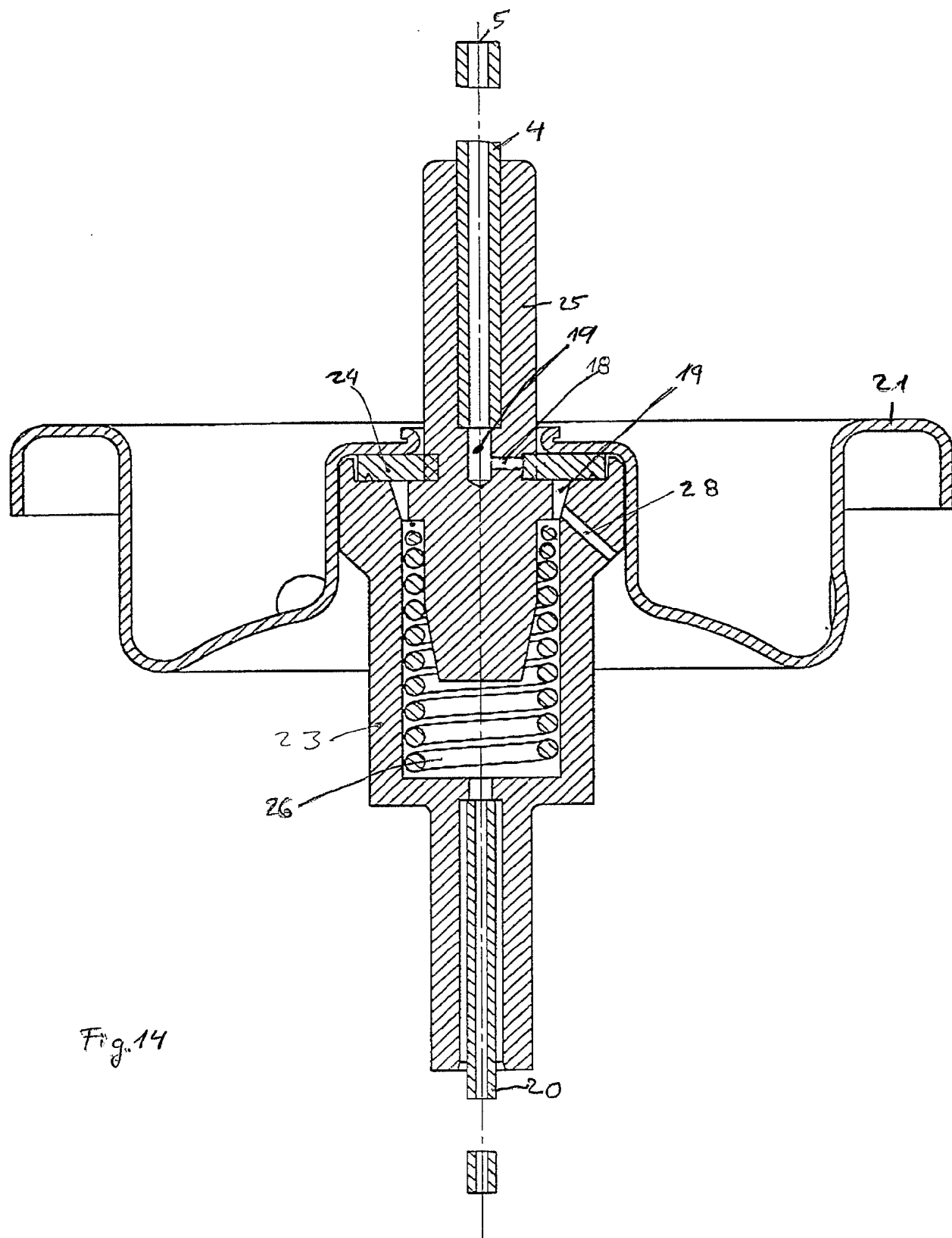
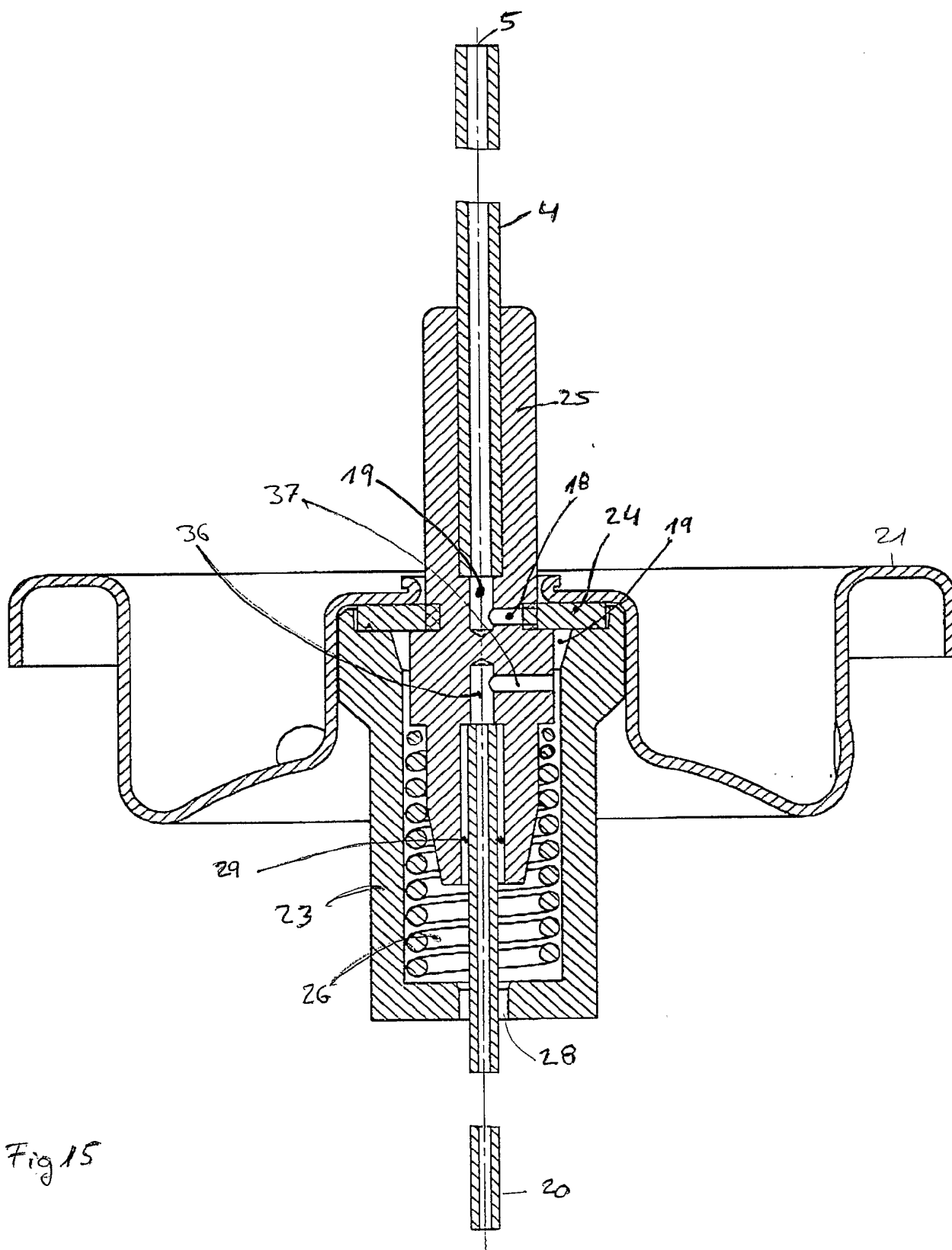
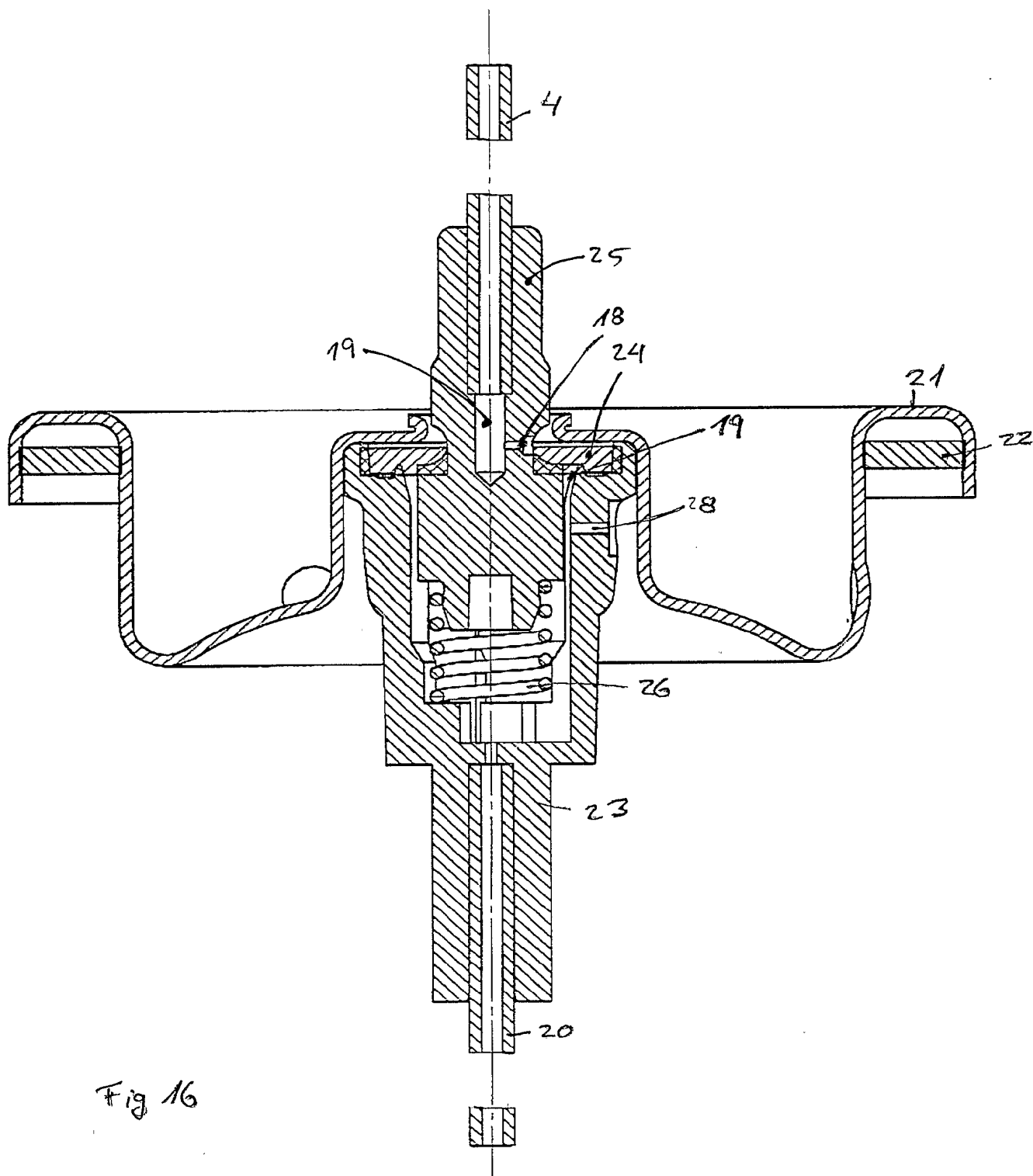
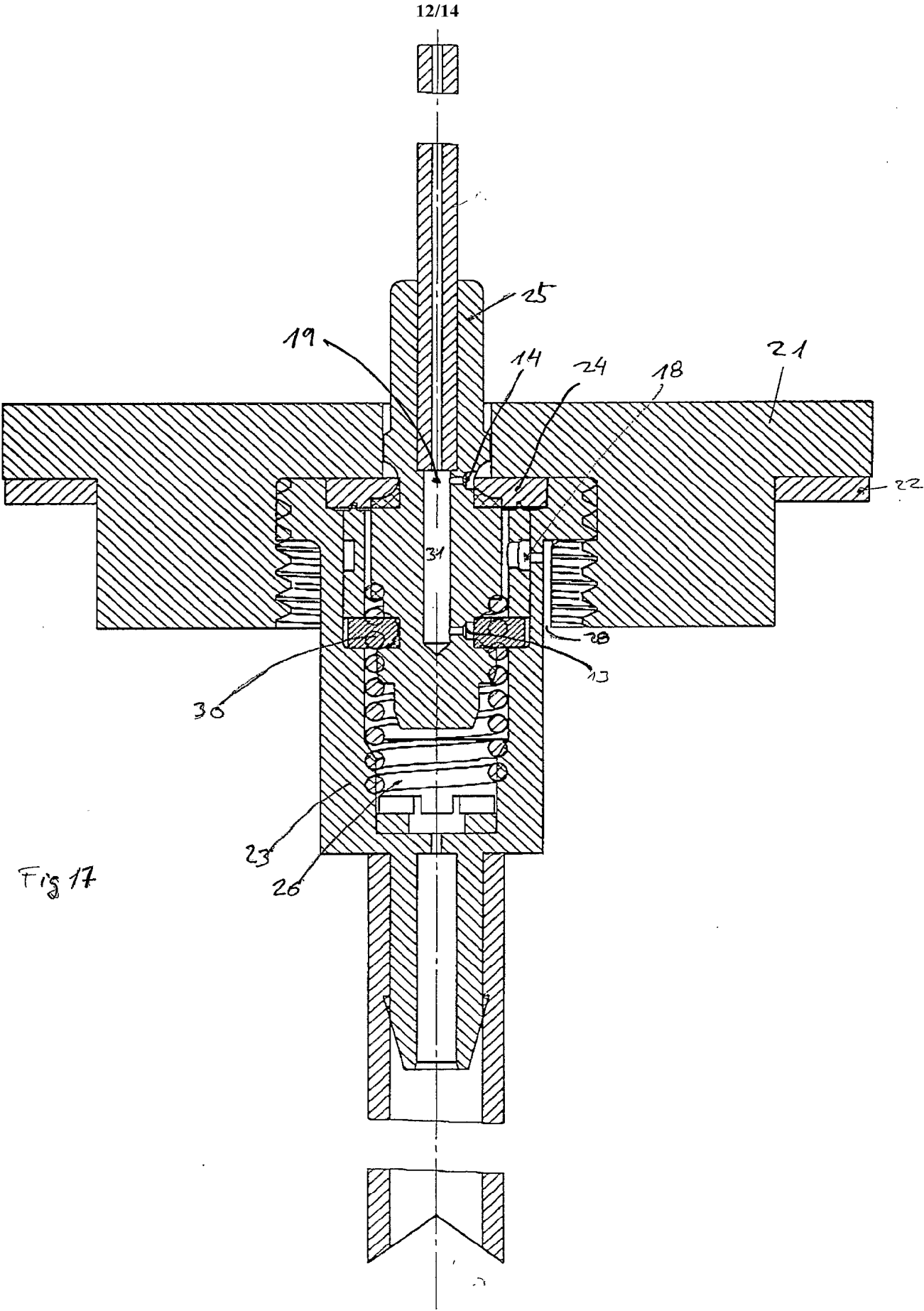


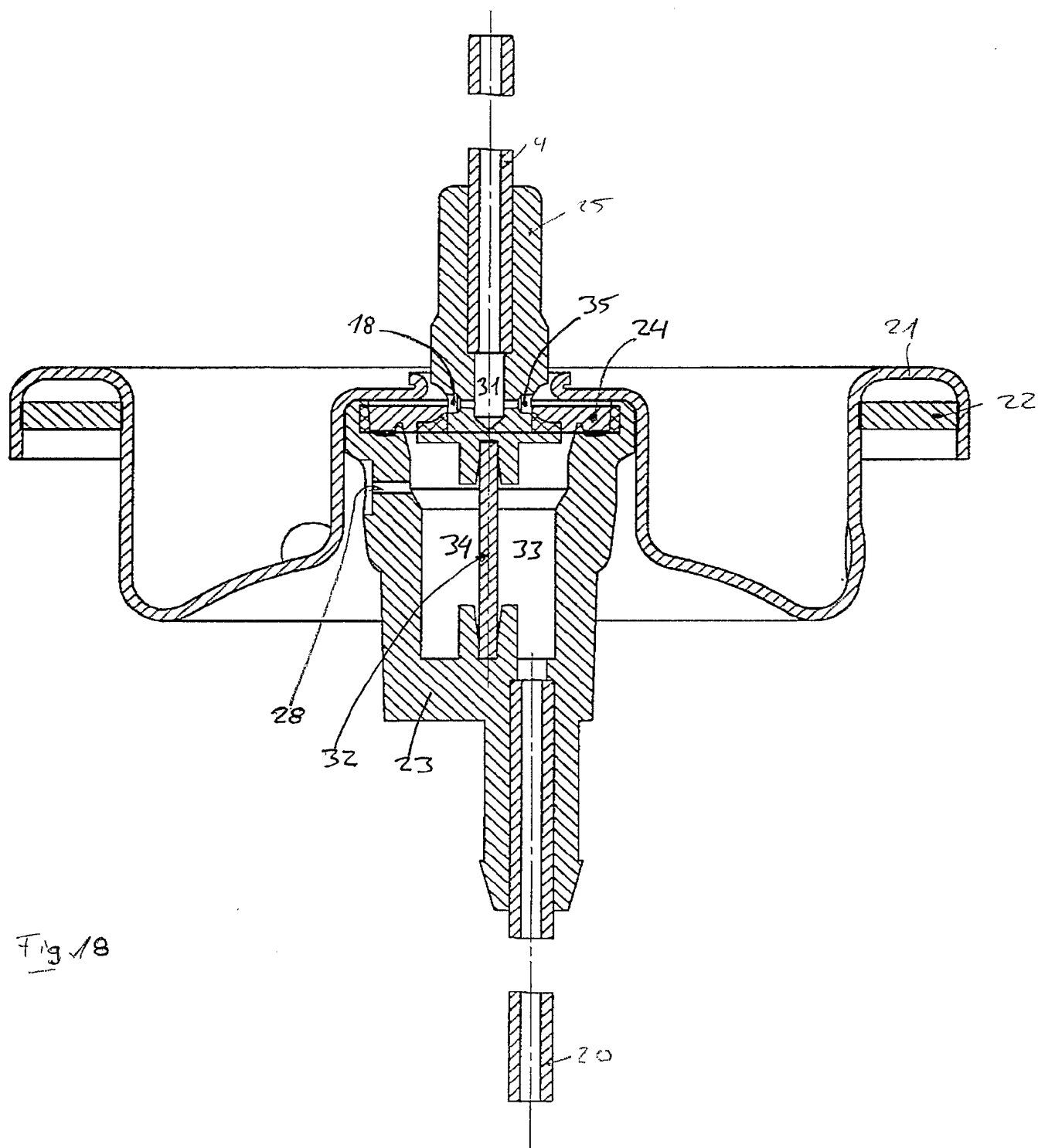
Fig 13











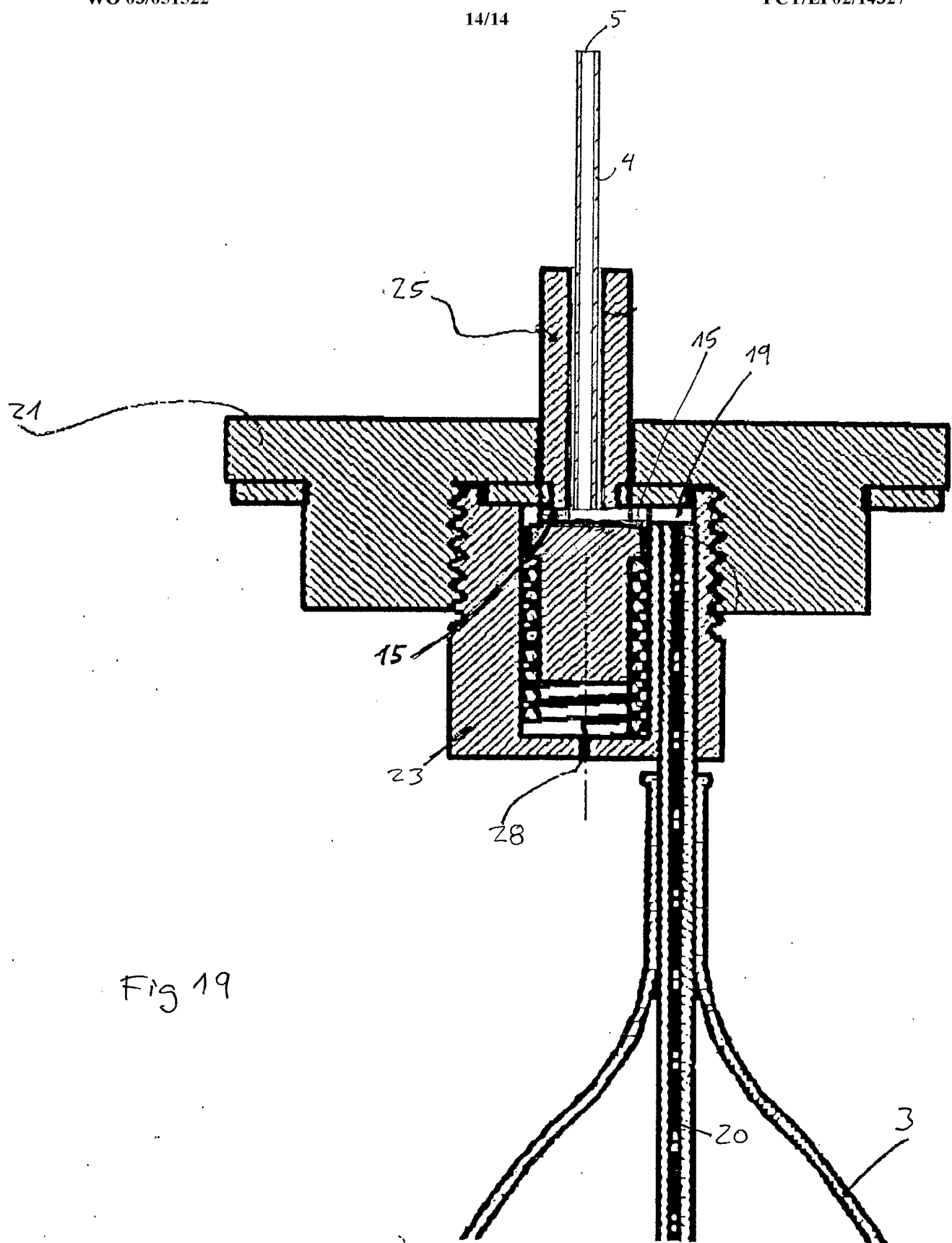


Fig 19